

Optical Damage Testing of Materials for use in 157nm Photo-lithographic Systems

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Abstract

As photolithographic processes utilize ever shorter wavelengths to produce more densely packed circuitry on silicon chips, the choice of materials suitable for use in the DUV spectral region becomes severely limited. We report here on preliminary life test results for calcium fluoride irradiated at 157nm by F₂ laser beams. The sample housing and beam delivery tubes were purged continuously with high purity nitrogen to keep the background oxygen level as low as possible and to sweep away any potential organic gases liberated from the sample mounting hardware and overall experimental apparatus. Data were collected to evaluate induced changes in transmission, wavefront distortion and birefringence over the course of billions of shots at a nominal fluence of 0.1 mJ / cm².

Richard G. Morton was educated at The Queen's University of Belfast, receiving the degrees B.Sc. Honours Physics (1970) and M.Sc. Optoelectronics (1972). Mr. Morton has extensive experience in the field of lasers / electrooptics. Most recently, Mr. Morton designed and executed the first International SEMATECH 193 nm materials testing programs at Cymer, Inc.

EXPERIMENTAL SETUP

Please refer to Figure 1, below.

The four CaF_2 samples, each measuring 2cm x 4cm in cross section and 8cm in length, are housed in an aluminum enclosure with a removable cover which can be clamped in place. An O-ring, which runs along the top edge of the enclosure, allows the cover to form a seal which, in turn, allows the interior of the enclosure to be purged with nitrogen. The O-ring is situated in such a fashion that exposure to scattered 157nm radiation is avoided. It is known that photodecomposition of hydrocarbons is a problem even at 248nm, and was assumed to be potentially far more serious at 157nm. Such decomposition could lead to contamination to sample surfaces and to attenuation of the 157nm laser beams within the enclosure. The only materials exposed to radiation in the sample housing are aluminum, stainless steel and the samples themselves. The only exception to this is when average power measurements are made with a conventional, wired detector head at intervals of several billion pulses.

A boil-off nitrogen purge rate of three liters-per-minute maintains the atmosphere around the samples at < 5ppm of O_2 and with H_2O as low as possible (generally <0.5ppmv). The nitrogen is purified with a catalysis filter which removes O_2 , H_2O and many organic contaminants, along with particles larger than 0.003 μm . This appears to be effective, since we have observed no discolorations on any of the parts within the sample housing, nor any visible deposits on the sample surfaces.

The samples are kinematically mounted to allow accurate relocation after removal for birefringence and interferometric measurements. Since calcium fluoride is much more susceptible to chipping than fused silica, care was taken in the mounting design to avoid contact with relatively sharp locating pins.

157nm radiation from two, 2kHz F_2 lasers, illuminates the samples from opposite directions, along the 8cm dimension axis. The laser pulses are timed to fire alternately at intervals of 250 microseconds, thus giving a true representation of 4kHz laser operation, and avoiding any possible issues with damage mechanisms that depend on the time between pulses. Previous experiments on optical damage at 193nm¹ were conducted by using delay lines to synthesize 8kHz pulse repetition rates, but the pulses were in bursts of 4, where the pulse spacing was about 50ns and the time between bursts of 4 pulses was 500 microseconds.

The 193nm¹ testing demonstrated that accelerated life-testing by increasing the energy density does not necessarily duplicate the low fluence, tens of billions of shots regimen experienced by real projection optics in a fabrication facility. In fact, a new phenomenon was discovered in which many types of fused silica exhibit "*rarefaction*"; which is in opposition to the better known "*compaction*" seen at higher fluence levels. This was yet another reason to conduct the accelerated life testing of calcium fluoride at 157nm irradiation wavelength by increasing the pulse repetition rate rather than the fluence per pulse. Although calcium fluoride is a crystalline material, and thus less likely to undergo unforeseen damage mechanisms akin to those that occur in fused silica, it was decided to adhere to the low fluence, increased pulse repetition rate paradigm while checking for radiation induced damage mechanisms.

The two 157nm laser beams were carefully aligned such that they are as collinear as possible. Illuminating the samples in this way simplifies the optical design, and results in a significantly more even distribution of intensity through the samples than unidirectional irradiation. Another feature of this arrangement is that there are no optical coatings in the beam delivery system except for those in the lasers. Coatings for beam splitting and combining are generally more prone to optical damage than those used at normal incidence. This problem is more pronounced as the operating wavelength becomes shorter, and the scope of the experiment did not include exposure of multi-layer dielectric coatings. We were looking purely at substrate material response to irradiation.

The laser energy is monitored using the internal energy monitors, and the pulse width is monitored using a vacuum photo-diode positioned to receive scattered light from within the sample housing. Energy and pulse-width is recorded for both lasers on a periodic basis along with the current O_2 and H_2O levels. The energy and pulse width information is used to calculate the dose delivered to the samples through the

integral-square definition of pulse-width. The units are $10^6((\text{mJ}/\text{cm}^2)^2)/\text{ns}$ according to the Sandstrom definition of integral square pulse width as it pertains to compaction dose:

$$t_{IS} = \frac{\left(\int T(t)dt\right)^2}{\int T^2(t)dt}$$

After a given number of pulses, the samples are tested for changes in transmission, optical path length, and birefringence. The transmission is tested by placing a pyro-electric detector in the enclosure, and measuring the pulse energy after it has passed through the exposed area of the samples, an unexposed area of the samples, and the area with the samples removed. the samples have been mounted on a sliding platform to accommodate this measurement. Changes in optical path length are checked using an interferometer, and changes in birefringence are checked using a scanning birefringence measurement system.

To date, measurements have been taken at 1 billion (B), 2B, and 4B pulses, and no measurable changes have been detected in the birefringence of the samples. The same is true of the interferograms; no refractive index gradients have been induced by irradiation at 157nm. The approximate dose delivered to the samples at a fluence in the $0.1 \text{ mJ}/\text{cm}^2$ range after 4 billion shots is $3.07 \times 10^6((\text{mJ}/\text{cm}^2)^2)/\text{ns}$.

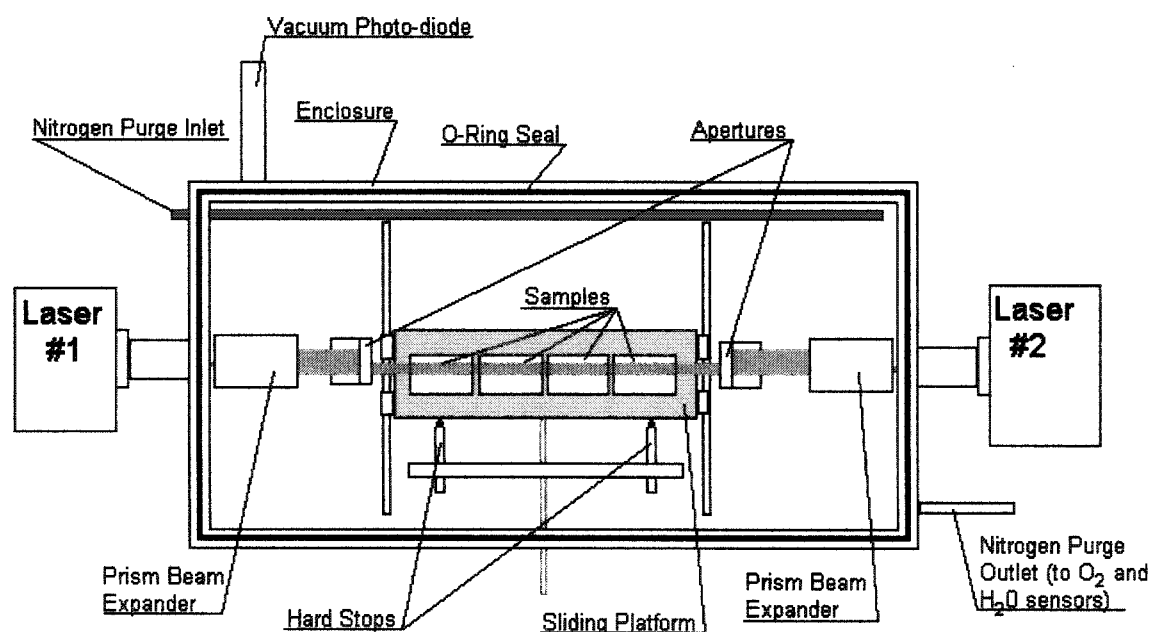
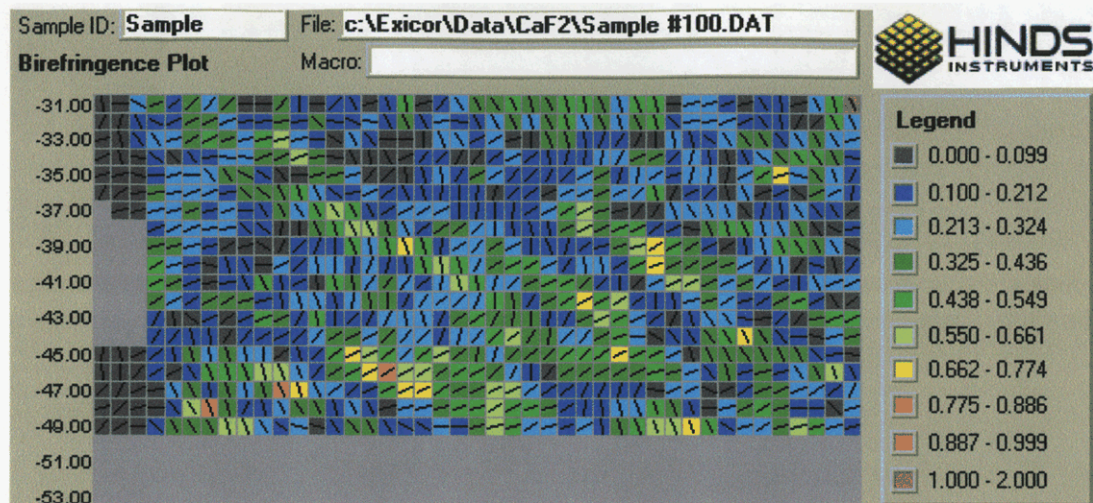


Figure 1 – 157nm Sample Exposure Setup

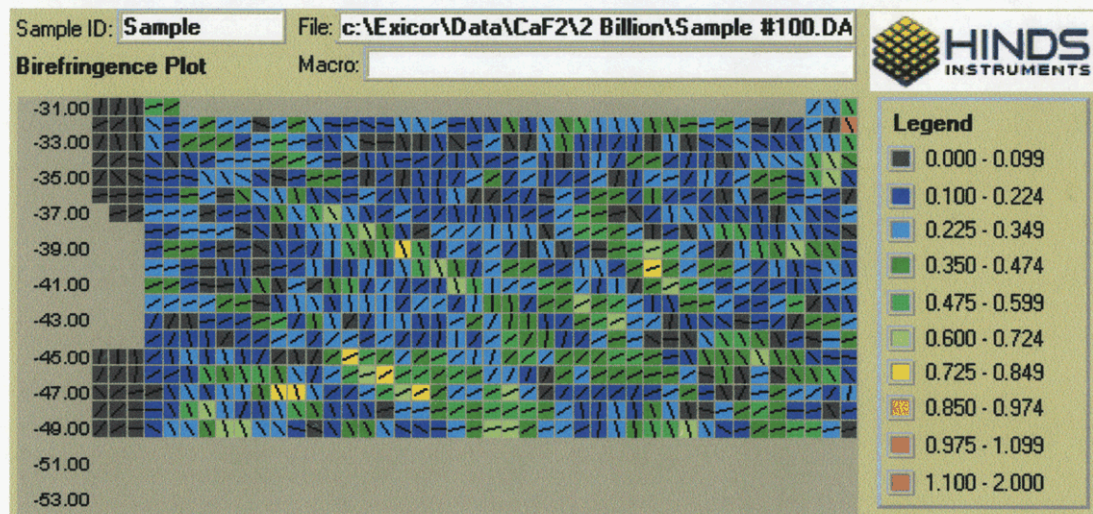
WAVEFRONT AND BIREFRINGENCE MEASUREMENTS

Figures 2 and 3 show birefringence plots for both samples. Plots for unexposed, 2 billion shots and 4 billion shots are presented for each sample. The units referred to in the legend to the right of each plot are in nm/cm, except for the initial condition map for sample #102, where the units are nm/8cm, the total length of the sample. The as-received average birefringence in the samples is about 0.2 nm/cm for sample#100; and zones of 0.2 average and 0.5 average nm/cm for sample#102. These are shown as color-coded regions in the spatially resolved birefringence plots.

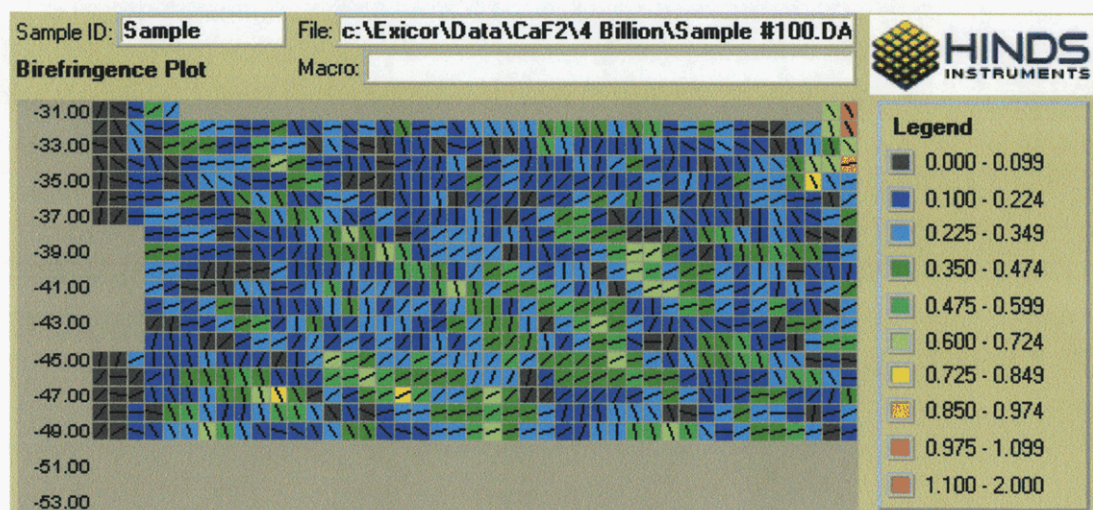
Figures 4 through 7 show interferograms for sample#100 at 1 and 4 billion shots , and sample #102 at 1 and 4 billion shots, sequentially. Note that sample #100 actually shows a slight improvement at 4 billion shots compared to 1 billion shots. At 4 billion shots the RMS wavefront distortion is 0.0125microns, and at 1 billion shots is 0.0157microns. It is possible that the difference is not caused by the 157nm radiation, but perhaps by miniscule differences in thermal gradients within the material at the time of tests. Sample #102 shows no measureable change , at a constant 0.0256 RMS microns wave front distortion.



Initial condition

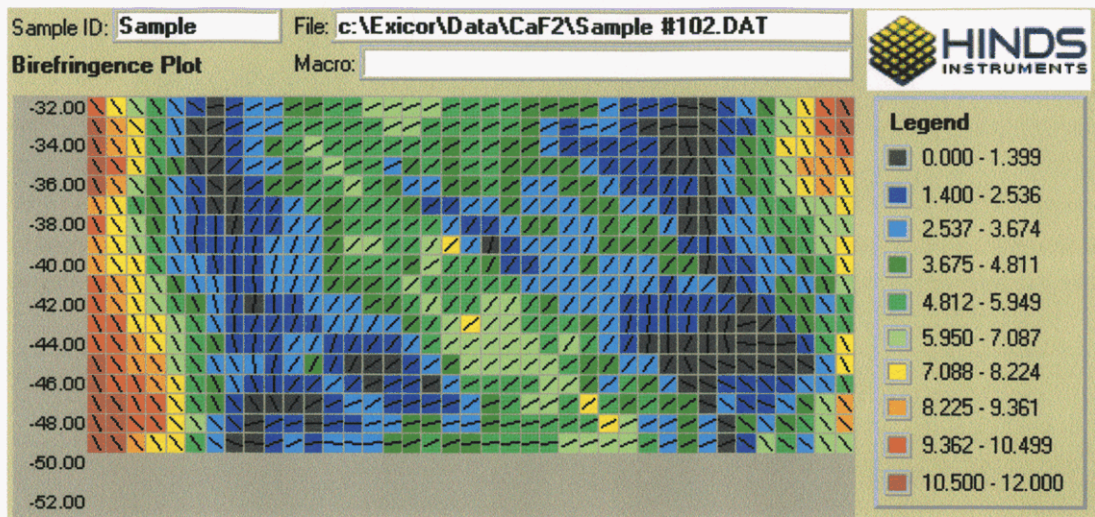


After 2 billion shots.

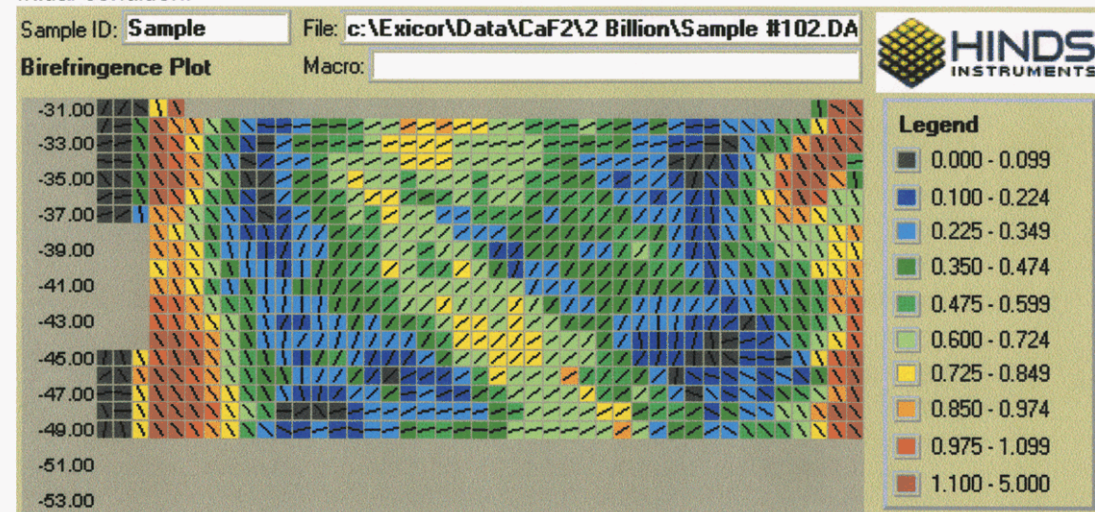


After 4 billion shots. SAMPLE # 100 Birefringence plots

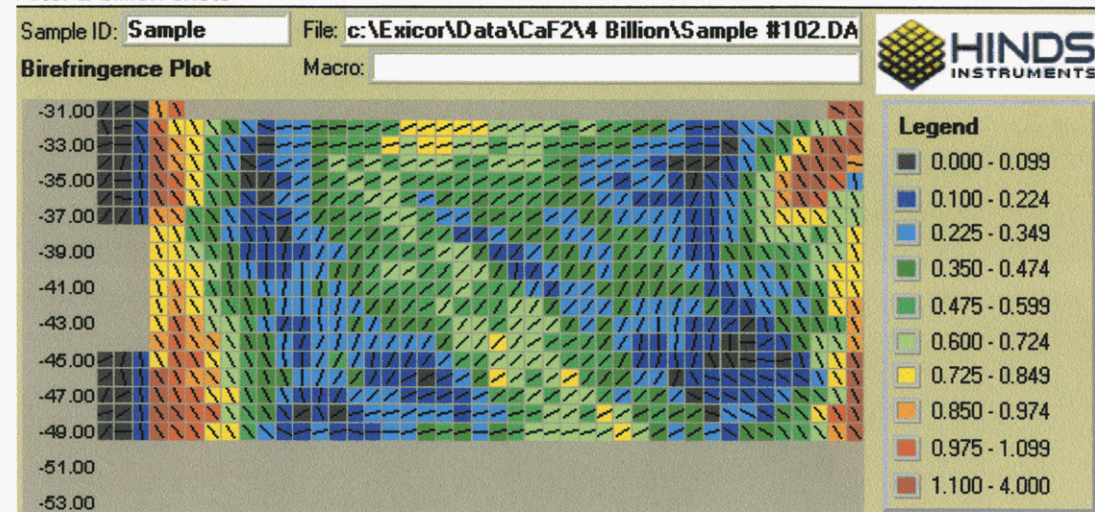
Figure 2 Birefringence plots for sample # 100



Initial condition.



After 2 billion shots



After 4 billion shots. SAMPLE # 102 Birefringence plots

Figure 3 Birefringence plots for sample # 102

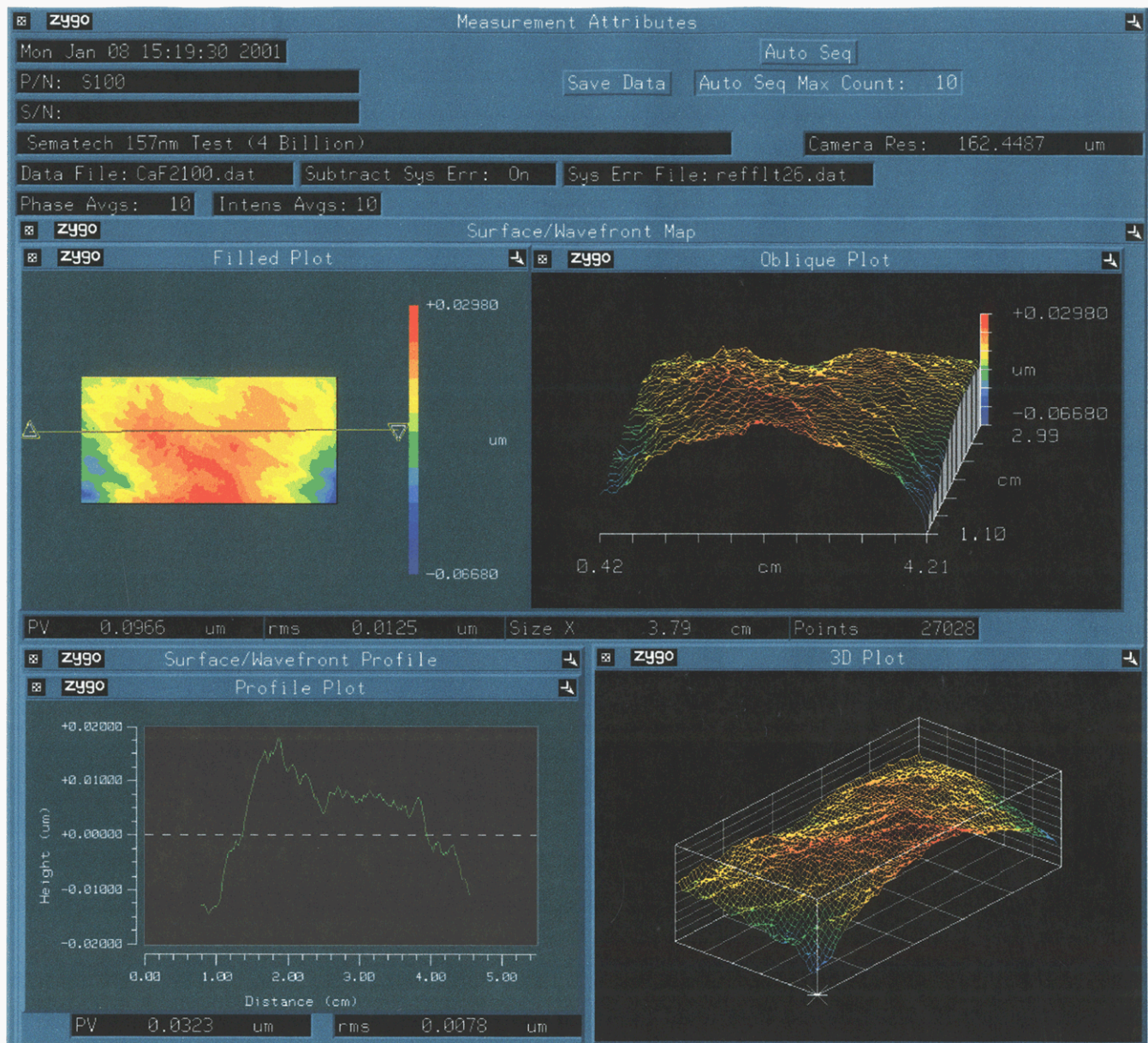


Figure 4. Sample #100 after 4 billion pulses

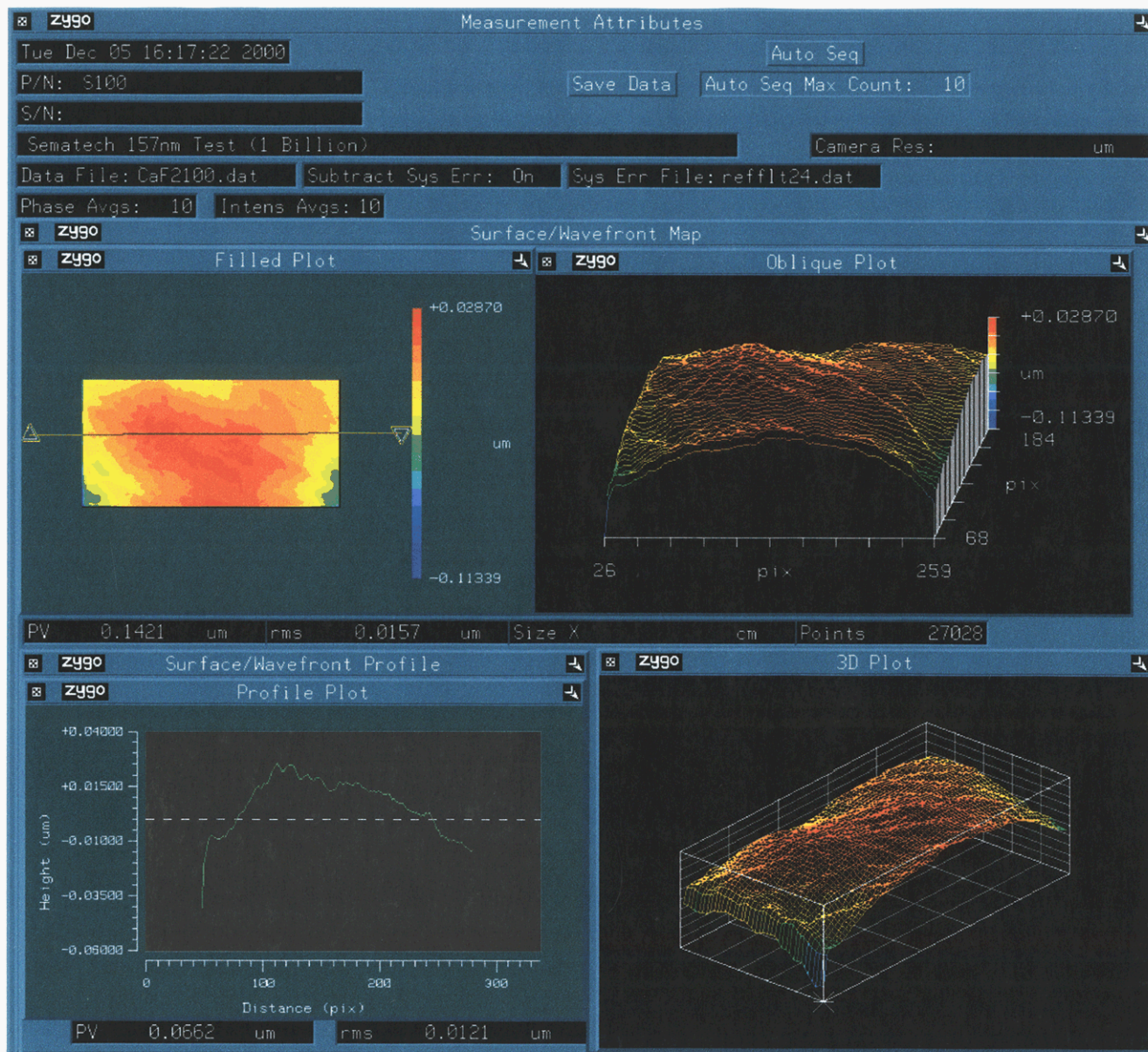


Figure 5. Sample #100 after 1 billion pulses

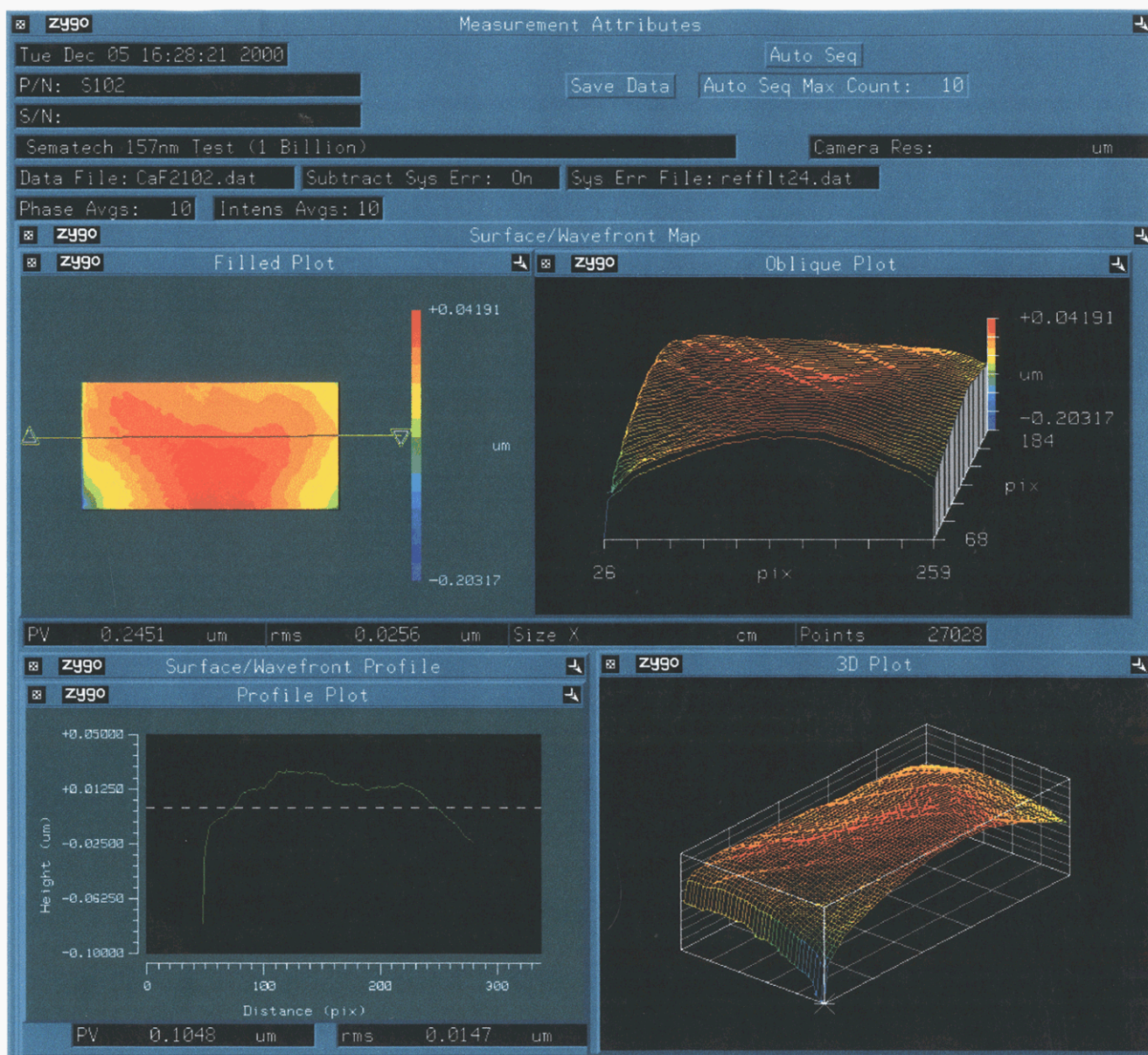


Figure 6. Sample#102 after 4 billion shots

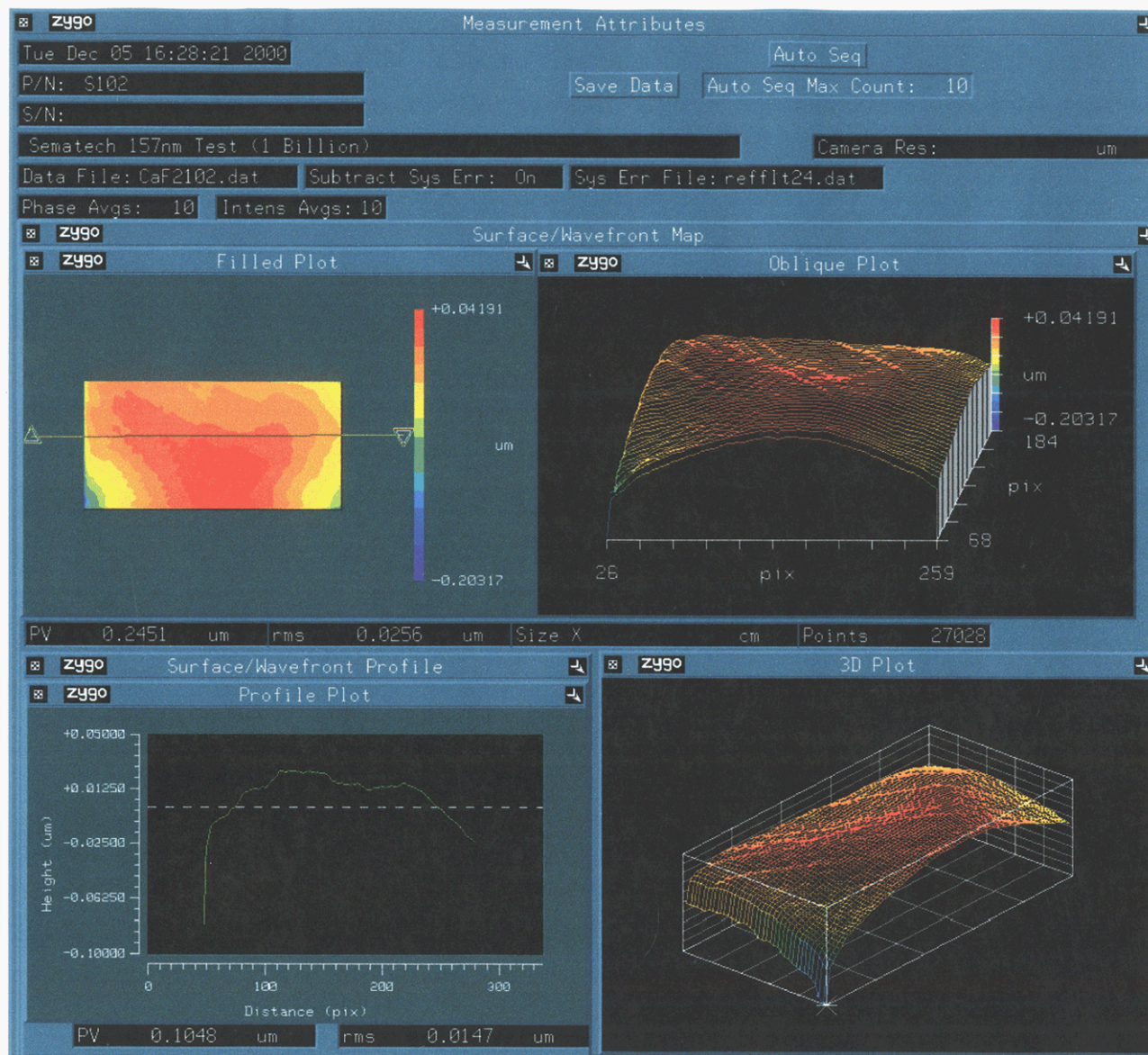


Figure 7. Sample #102 after 1 billion shots.

ABSORPTION MEASUREMENTS

Although sample absorption measurements were made in situ, these were not sample-resolved, meaning that the transmission of all 4 samples in series was periodically recorded. Another difficulty with the on-line measurements was that they were made after exposing the samples to air, and thus offset by unknown amounts due to deposition of water and hydrocarbons. These measurements were not intended to show small changes in transmission, but rather to monitor any gross changes as a result of irradiation at 157 nm.

A much more sophisticated set of measurements on the individual samples were conducted at MIT Lincoln Laboratory, which is also performing research in support of International SEMATECH objectives. The samples were removed from the exposure facility after 10 billion pulses and sent to MIT Lincoln Laboratory for the absorption measurements.

Transmission measurements were made using a Varian spectrophotometer load-locked to a 172nm lamp-based cleaning station. Prior to measurement, the samples were inserted into the cleaning station for 20 minutes and exposed to 172nm irradiation in a nitrogen atmosphere with 1% O₂ content. Photo-assisted oxidation is very effective in destroying and removing hydrocarbon contaminants which may be deposited on the sample surfaces from environmental sources. After cleaning, the sample is moved through a load lock into the Varian spectrophotometer, which has a pure nitrogen atmosphere.

This UVO cleaning technique has proven effective in removing adsorbed hydrocarbon compounds. Tests have shown that a monolayer of hydrocarbon attenuates 157nm irradiation by approximately 1%, thus it is critical that samples be properly cleaned prior to measurement.

The results for the two sample measured, International SEMATECH #100 and #102 are given below.

Sample #100.

The theoretical Fresnel reflection loss is 9.5% for two surfaces. The bulk and scattering losses total 1.8%, measured at 157nm wavelength. If the entire loss is ascribed to bulk absorption, the calculated absorption for this material would be 0.0011 cm⁻¹. The industry-wide target value is presently about 0.002 cm⁻¹, according to researchers in the field at MIT Lincoln Laboratory. After irradiation at 157nm for 10 billion shots, this sample has an acceptable coefficient of absorption. In addition, there has been no change induced by the radiation, which can be seen in Figure 8.

Sample #102.

For this sample there are three observations of interest. The first is that the calculated absorption for this material would be 0.0039 cm⁻¹, based on the same assumptions applied to Sample#100. This places the sample "out of specification" after 10 billion shots. The second observation is that this sample exhibited an increased absorption after irradiation, in contrast to Sample#100, which did not. The 157nm wavelength absorption increased by about 1% over the 8cm beam path-length as a result of the exposure. The third observation is that Sample#102 started out with lower transmission than Sample#100. The initial transmissions were 87.2% and 85%, respectively. Figure 9 shows the transmission curves for sample#102.

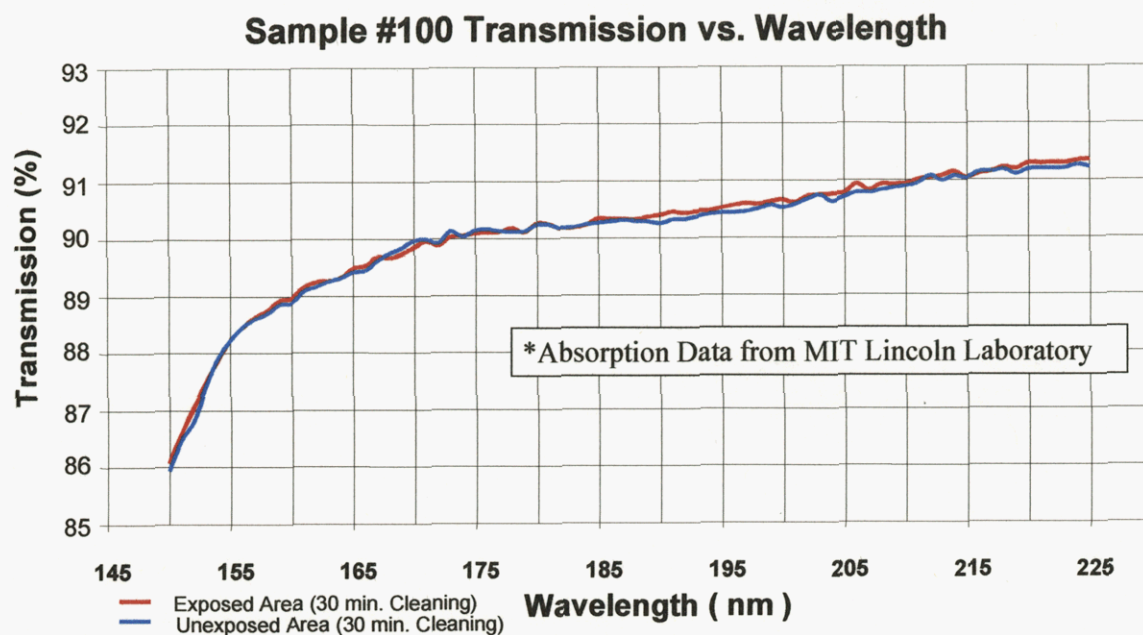
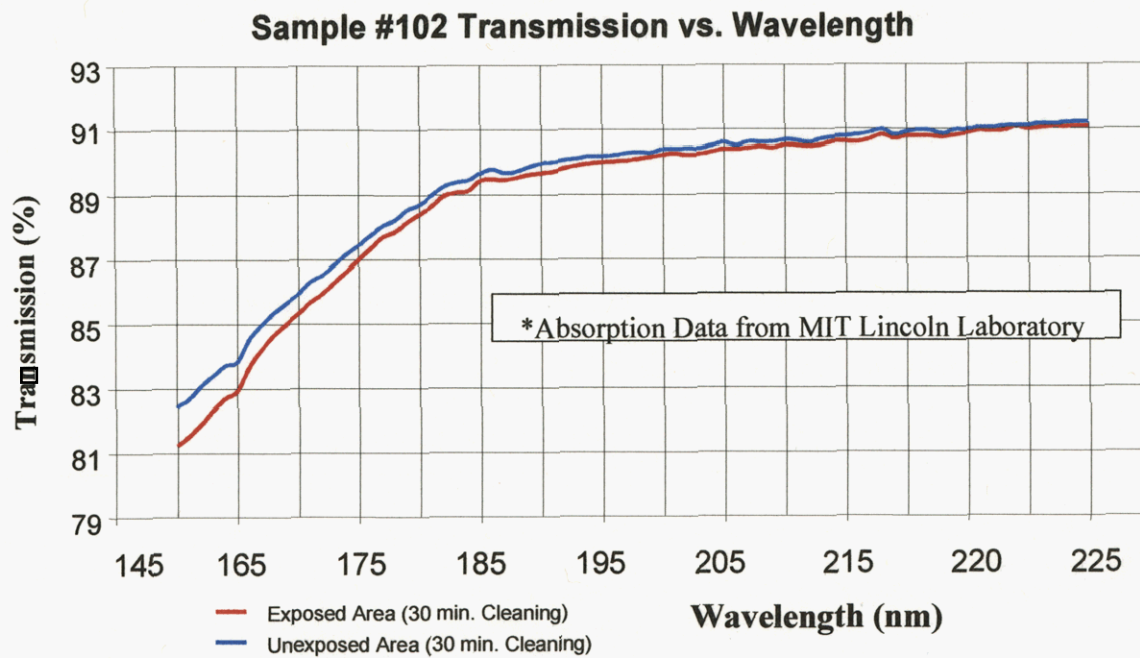


Figure 8. Absorption curves for sample#100 (above)

Figure 9. Absorption curves for sample#102 (below)



CONCLUSIONS

As of the shot count reported in this document (4 billion), there have been no significant changes in birefringence or wave front distortion in the irradiated calcium fluoride samples as compared to their initial states. *(Update: as of 10 billion pulses, the above statement remains true. March 12, 2001)* Changes in transmission appear to be dependent on the initial transmission of the material samples, which is indicative of a low level impurity issue in manufacture.

Based upon the above statement, it seems likely that carefully process-controlled calcium fluoride materials will be capable of maintaining their initial optical properties over anticipated service life times of tens-of-billions of shots.

The intended duration of the exposure is 50 billion shots at the 0.1mJ/cm² level , which is representative of several years of operation of projection optics. Upon completion of 50 billion shots, a final report will be issued by International SEMATECH , containing the results from this "marathon" exposure. It is anticipated that at least one sample will be replaced during the course of the exposure testing so that another, with different initial properties can be evaluated. The projected time for completion of the 50 billion shot exposure is August of 2001.

ACKNOWLEDGEMENTS

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